

# APPENDIX B

## Audit Checklists

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# BIOCHEMICAL OXYGEN DEMAND: METHOD EVALUATION CHECKLIST

## Method 5210-B, Standard Methods, 18th ed.

Laboratory Name: \_\_\_\_\_ Lab Director: \_\_\_\_\_

Date: \_\_\_\_\_

Evaluator: \_\_\_\_\_

### I. EQUIPMENT

#### A. Dissolved Oxygen Measurement

- ☐ 1. The laboratory measures dissolved oxygen by:
  - ☐ Winkler titration
  - ☐ D.O. meter and probe
- ☐ 2. Calibration and maintenance of D.O. Meter:
  - ☐ Calibration against air of known DO concentration.
  - ☐ Calibration against water of known DO concentration (determined by iodometric method).
  - ☐ Meter zeroed with saturated sodium sulfite solution.
  - ☐ Electronic zero checked.
  - ☐ Manufacturer's procedure followed exactly.
  - ☐ Membrane is replaced as necessary, and a record of maintenance kept.
  - ☐ Air bubbles are excluded from under or around membrane when reading meter.
  - ☐ The sample is adequately mixed during measurement.
- ☐ 3. Winkler Titration
  - ☐ Cross contamination of Winkler reagents is prevented; air bubbles are excluded.
  - ☐ KI and Manganous Sulfate precipitate is allowed to settle.
  - ☐ After addition of 1 mL conc. sulfuric acid, solution is mixed until precipitate is dissolved.
  - ☐ The sample is titrated to a pale yellow straw color using a starch indicator.

#### B. Incubator

- ☐ 1. The incubator temperature is kept at  $20 \pm 1^\circ \text{C}$  when in use.
- ☐ 2. The lab keeps a record of incubator temperature.
- ☐ 3. The lab keeps a record of incubator maintenance.
- ☐ 4. Samples are incubated in the dark.

#### C. Glassware

- ☐ 1. The laboratory uses bottles of 250-300 mL capacity, with ground glass stoppers.
- ☐ 2. Large bore volumetric pipets are used for samples directly pipetted into BOD bottles.
- ☐ 3. Glassware is available for making dilutions. (ie, 1:1, 1:10, etc.).
- ☐ 4. BOD bottles are cleaned with a detergent, rinsed thoroughly and drained before used.
- ☐ 5. BOD bottles are rotated, i.e. used to hold different samples after each analysis.

### II. REAGENTS

#### A. 0.025 N Sodium Thiosulfate or 0.025 N Phenylarsine Oxide Titrant (Winkler Method) is:

- ☐ 1. Purchased commercially, or
- ☐ 2. Prepared in the lab by dissolving 6.205 g  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  and 0.4 g NaOH in 1L distilled
- ☐ 3. Standardized with potassium bi-iodate solution before use (if prepared in lab).
- ☐ 4. Documented when standardized (if prepared in lab).

#### B. Standard potassium bi-iodate (Winkler Method)

- ☐ 1. 0.021M solution: dissolve 812.4 mg  $\text{KH}(\text{IO}_3)_2$  in distilled water and dilute to 1 L.
- ☐ 2. Used for the standardization of sodium thiosulfate.

#### C. Starch Indicator Solution (Winkler Method):

- ☐ 1. Prepared in the laboratory
- ☐ 2. Purchased commercially

D. Neutralizing and dechlorinating reagents:

- ☐ 1. Fresh  $\text{Na}_2\text{SO}_3$  is prepared daily for neutralizing chlorine (chlorinated effluents only).
- ☐ 2. 1 N  $\text{H}_2\text{SO}_4$  for neutralizing high pH samples.
- ☐ 3. 1 N NaOH for neutralizing low pH samples.

E. Nutrients for dilution water:

- ☐ 1. Prepared in laboratory:(list) \_\_\_\_\_
- ☐ 2. Purchased commercially:(list) \_\_\_\_\_
- ☐ 3. Discarded if there is any sign of biological growth in stock bottle.
- ☐ 4. The pH of the phosphate buffer solution is 7.2.

F. Dilution water is:

- ☐ 1. Made from: ☐ Distilled ☐ Deionized ☐ Other(specify) \_\_\_\_\_.
- ☐ 2. Equilibrated to 20° C when in use.
- ☐ 3. Protected by using clean glassware, tubing and bottles.
- ☐ 4. Saturated with oxygen before use: ☐ By shaking ☐ By filtered aeration.
- ☐ 5. Stored with a permeable plug.
- ☐ 6. Prepared by adding 1 mL of each of nutrient solution per liter of water.

### III. PROCEDURE

A. Pretreatment Steps

- ☐ 1. Are samples checked for pH?
- ☐ 2. If samples contain caustic alkalinity or acidity, are they:
  - ☐ Neutralized?
  - ☐ Seeded?
- ☐ 3. If samples were chlorinated, are they:
  - ☐ Checked for chlorine?
  - ☐ Neutralized with  $\text{Na}_2\text{SO}_3$  when present?
  - ☐ Seeded?
- ☐ 4. Are chlorinated and industrial effluents seeded even though no residual chlorine detected?

B. Transfer & Dilution Steps

- ☐ 1. Are samples well mixed before pipetting?
- ☐ 2. Are samples warmed to 20 C before pipetting? (If were in cooler)
- ☐ 3. Is sample prepared within 2 hours of collection if not cooled?
- ☐ 4. Are at least two dilutions set up per sample? (3 is preferable)
- ☐ 5. Are dilution water blanks set up for each analysis day and for each bottle of dilution water?
- ☐ 6. Are sample volumes less than 3 mL diluted in a graduated cylinder or volumetric flask before making final dilution in the BOD bottle?
- ☐ 7. Are additional nutrients added to bottles when using a small volume of dilution water?

C. Seed

- ☐ 1. Are seed controls set up when samples are seeded?
- ☐ 2. Is the seed:
  - ☐ added to the dilution water?
  - ☐ added directly to BOD bottle?
  - ☐ never used, except for GGAs, blind standards and reference samples?
- ☐ 3. Is the seed made from:
  - ☐ an unchlorinated, non-toxic effluent from the STP or,
  - ☐ a supernatant of settled raw wastewater?
  - ☐ other(specify)\_\_\_\_\_.
- ☐ 4. Do at least two seeded blanks deplete at least 2 mg/L with a residual of 1 mg/L after 5 days? If not,
- ☐ 5. Does lab the lab calculate the seed correction factor with only those bottles meeting the depletion

D. Initial DO & Bottle Status

- ☐ 1. Is an IDO measured on every sample dilution, after set up and before incubation?

- ☐ 2. If an IDO > 9 mg DO/L at 20 C, is sample stripped of excess DO by agitation or aeration?
- ☐ 3. Does analyst insure that no air bubbles are present in BOD bottle before incubation?
- ☐ 4. Are water seals on bottles protected to prevent drying?
- ☐ 5. Are BOD bottles labeled for sample ID?

E. Five Day Residual DO

- ☐ 1. Is the residual DO after 5 days at least 1 mg/L?
- ☐ 2. Is the depletion after 5 days at least 2 mg/L?
- ☐ 3. Are BOD calculations done correctly?
- ☐ 4. Are seed corrections used correctly?

### III. QUALITY CONTROL/QUALITY ASSURANCE AND DATA

A. Known Standard Used:

- ☐ 1. Laboratory prepared glucose-glutamic acid
  - ☐ Are reagents dried for at least 1 hour at 103° C?
- ☐ 2. Commercially prepared glucose-glutamic acid
- ☐ 3. Other(specify) \_\_\_\_\_

B. Control Limits:

- ☐ 1. Do the 5 day BODs for the GGAs fall within established control limits ( $198 \pm 30.5$  mg/L)?
  - ☐ 2. Are limits established for replicates?
  - ☐ 3. Do the 5 day BODs for other standards fall within established control limits?
- List these standards and their limits: \_\_\_\_\_

C. Data Reporting and Interpretation

- ☐ 1. Is the DO uptake of dilution water blanks always 0.2 mg DO/L or less?
- ☐ 2. Are corrective actions taken when DO uptake of dilution water blanks exceeds 0.2 mg DO/L?
- ☐ 3. Do at least two sample dilutions deplete at least 2 mg/L with a residual of 1 mg/L?
  - ☐ If not, does the lab change the dilutions used to meet this criteria?
- ☐ 4. Is the average BOD calculated using only those bottles meeting the depletion criteria?
- ☐ 5. What BOD value is reported when duplicates are set:
  - ☐ The highest
  - ☐ The lowest
  - ☐ The average
  - ☐ Other (specify): \_\_\_\_\_
- ☐ 6. Does the laboratory experience sliding BODs? If yes:
  - ☐ How they are reported? (ave., high, low) \_\_\_\_\_
  - ☐ Are results qualified as sliding?
  - ☐ Does the lab change sample volume to reduce the effect of toxics on results?

**AMMONIA NITROGEN PREDISTILLATION:  
METHOD EVALUATION CHECKLIST  
(Method 4500-NH<sub>3</sub> B, Standard Methods, 18th ed.)**

Laboratory Director: \_\_\_\_\_ Laboratory Name: \_\_\_\_\_

Date: \_\_\_\_\_ Evaluator: \_\_\_\_\_

**I. EQUIPMENT**

- ☐ A. Distillation Apparatus
  - ☐ A Kjeldahl or Micro Kjeldahl distillation apparatus or a borosilicate flask of 800-2000 mL capacity
  - ☐ A vertical condenser with outlet tip
  - ☐ 500 mL Erlenmeyer flasks for collecting distillate
- ☐ B. A pH meter or short range pH paper
- ☐ C. Volumetric pipets and flasks

**II. REAGENTS**

- ☐ A. Ammonia-free water is used for dilution of samples and preparation of all reagents and standards.
  - ☐ Prepared by ion exchange.
  - ☐ Prepared by distillation.
- ☐ B. Borate buffer solution is:
  - ☐ Purchased commercially.
  - ☐ Prepared in the laboratory by making a 0.025 M sodium tetraborate solution by dissolving either 9.5 g of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>•10H<sub>2</sub>O or 5.0 g anhydrous Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> in 1 L with water then adding 88 mL of 0.1M NaOH to 500 mL of the 0.025 M Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> solution and diluting to 1 L.
- ☐ C. Sodium Hydroxide, 6 N
  - ☐ Purchased commercially
  - ☐ Prepared in lab by dissolving 240 g NaOH in water, cooling and diluting to 1 L
- ☐ D. Dechlorinating Reagent for residual chlorine
  - ☐ 1. Never needed
  - ☐ 2. Sodium Sulfite, prepared fresh daily by dissolving 0.9 g Na<sub>2</sub>SO<sub>3</sub> in water and diluting to 1 L.
  - ☐ 3. Sodium Thiosulfate, prepared fresh weekly by dissolving 3.5 g Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>•5 H<sub>2</sub>O in 1 L dist. water.
  - ☐ 4. Phenylarsine Oxide, prepared by dissolving 1.2 g C<sub>6</sub>H<sub>5</sub>AsO in 200 mL 0.3 N NaOH, filtering if necessary and diluting to 1 L.
  - ☐ 5. Sodium Arsenite, prepared fresh weekly by dissolving 0.93 g NaAsO<sub>2</sub> in water and diluting to 1 L.
  - ☐ 6. Purchased commercially (List type and concentration) \_\_\_\_\_
- ☐ E. Neutralizing Agents
  - ☐ 1 N NaOH
  - ☐ 1 N H<sub>2</sub>SO<sub>4</sub>
- ☐ F. Indicating Boric Acid Solution
  - ☐ Purchased commercially
  - ☐ Prepared monthly in lab by dissolving 20 g H<sub>3</sub>BO<sub>4</sub> in water and diluting to 1 L
- ☐ G. Receiving Flask Solution is:
  - ☐ 1. 0.04 N H<sub>2</sub>SO<sub>4</sub> for the phenate and ammonia-selective electrode methods (1 mL conc. H<sub>2</sub>SO<sub>4</sub> diluted to 1 L with distilled water).
  - ☐ 2. Plain Boric Acid for the Nesslerization method, 50 ml in 500 ml flask.

- \_\_\_ 3. Indicating Boric Acid for the titrimetric method, 50 ml in 500 ml flask, made from:
  - \_\_\_ 3a. mixed Indicator Solution (200 g methyl red in 100 mL 95% ethyl or isopropyl alcohol combined with 100 mg methylene blue dissolved in 50 mL alcohol).
  - \_\_\_ 3b. 20 g  $\text{H}_3\text{BO}_3$  dissolved in water and 10 mL mixed indicator solution diluted to 1 L with distilled water.
- \_\_\_ H. Nessler Reagent:
  - \_\_\_ 1. 100 g  $\text{HgI}_2$  and 70 g KI are dissolved in water, added slowly to a cooled solution of 160 g NaOH in 500 mL water and diluted to 1 L
  - \_\_\_ 2. Replaced annually
  - \_\_\_ 3. Stored out of sunlight

### III. PROCEDURE

- \_\_\_ A. Steaming out distillation apparatus
  - \_\_\_ 1. 500 mL of water (and 20 mL borate buffer) are added to the distillation flask
  - \_\_\_ 2. The pH is adjusted to 9.5 with 6 N NaOH
  - \_\_\_ 3. Glass beads or boiling chips (that were rinsed with dilute NaOH) are added to flask
  - \_\_\_ 4. Distillation is carried out until no ammonia is in distillate, checked with Nessler reagent
- \_\_\_ B. Sample Preparation
  - \_\_\_ 1. STD. METHODS Procedure:
    - \_\_\_ 500 mL of sample or a volume diluted to 500 mL is neutralized to approximately pH 7, if necessary.
    - \_\_\_ 25 mL Borate Buffer is added to the sample and the pH is adjusted to 9.5 with 6 N NaOH.
  - \_\_\_ 2. EPA Procedure:
    - \_\_\_ 400 mL of sample or a volume diluted to 400 mL is adjusted to pH 9.5 with 1 N NaOH.
    - \_\_\_ 25 mL of Borate Buffer is added to the sample
- \_\_\_ C. Distillation
  - \_\_\_ 1. Distillation apparatus is left assembled after steaming out until sample is added
  - \_\_\_ 2. 50 mL of the appropriate acid is placed in the receiving flask
  - \_\_\_ 3. The tip of the delivery tube is placed below the surface of the acid receiving solution
  - \_\_\_ 4. The collection of 200 mL of distillate takes about 20-30 minutes
  - \_\_\_ 5. The collection of 300 mL of distillate takes about 30-50 minutes
  - \_\_\_ 6. The distillate is diluted to 500 mL
  - \_\_\_ 7. If the phenate method is used, the distillate is neutralized with 1 N NaOH before diluting to 500 mL
  - \_\_\_ 8. Concentrations above 1 mg  $\text{NH}_3\text{-N/L}$  are determined titrimetrically or by the ammonia selective electrode method
  - \_\_\_ 9. Concentrations below 1 mg  $\text{NH}_3\text{-N/L}$  are determined colorimetrically or by the ammonia selective electrode method
  - \_\_\_ 10. Blanks, standards, duplicates and spikes are distilled and analyzed the same as the samples

**AMMONIA NITROGEN, ION SELECTIVE ELECTRODE:  
EPA METHOD 350.3 EVALUATION CHECKLIST  
(Methods 4500-NH<sub>3</sub> F & G, Standard Methods 18th ed.)**

Laboratory Name: \_\_\_\_\_  
Date: \_\_\_\_\_

FID: \_\_\_\_\_  
Evaluator: \_\_\_\_\_

**I. EQUIPMENT**

- ☐ A. Meter and Probe
  - ☐ Electrometer (pH meter) with an expanded millivolt scale or,
  - ☐ Specific Ion Meter with a direct concentration readout
  - ☐ Ammonia Selective Electrode (Orion model 95-10 or equivalent)
- ☐ B. Magnetic Stirrer and Teflon Stir Bar
- ☐ C. Glassware
  - ☐ Volumetric pipets and flasks
  - ☐ 150 mL beakers, graduated cylinder

**II. REAGENTS**

- ☐ A. Ammonia-free water is:
  - ☐ Prepared by passing distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.
  - ☐ Prepared by adding 1 mL concentrated sulfuric acid or chlorine to distilled water and distilling .
  - ☐ Replaced when high blank values are obtained.
  - ☐ Protected from atmospheric contamination.
  - ☐ Used to dilute all standards, reagents and samples.
- ☐ B. Sodium Hydroxide (NaOH), 10 N
  - ☐ Prepared in lab by dissolving 400 g NaOH in 800 mL ammonia-free water, cooling and diluting to 1 L. (Or same proportions of NaOH and water)
  - ☐ Purchased commercially.
- ☐ C. Stock Ammonium Chloride Solution (NH<sub>4</sub>Cl), 1000 mg NH<sub>3</sub>-N/L (1.00 mL = 1.00 mg N)
  - ☐ Purchased commercially.
  - ☐ Prepared in laboratory: dried at 100° C before weighing 3.189 g anhydrous NH<sub>4</sub>Cl per liter water.
- ☐ D. Standard Ammonium Solution (NH<sub>4</sub>Cl), 10 mg NH<sub>3</sub>-N/L (1.00 mL = 0.01 mg N).
  - ☐ Purchased commercially.
  - ☐ Made from 10 mL Stock Ammonium Chloride (1000 mg NH<sub>3</sub>-N/L) in 1 L water.
  - ☐ Made from 10 mL of 100 mg NH<sub>3</sub>-N/L standard solution in 100 mL water.

**III. PROCEDURE**

- ☐ 1. Working standards are prepared by diluting stock or standard solutions with volumetric glassware.
- ☐ 2a. If an electrometer is used:
  - ☐ A tenfold change of NH<sub>3</sub>-N concentration produces a potential change of approximately 59 mV.
  - ☐ The meter is calibrated using 3 standards and a blank.
  - ☐ The standard curve is plotted on semi-log paper with mg NH<sub>3</sub>-N/L on the log axis and millivolts on the linear axis.
- ☐ 2b. If a Specific Ion Meter is used:
  - ☐ The meter is calibrated using at least two standards and a blank.
  - ☐ The standards bracket the concentration range of interest.
- ☐ 3. The temperature of standards and samples remain the same during calibration and testing.



- \_\_\_ 4. The same mixing rate is maintained for samples and standards.
- \_\_\_ 5. If a specific ion meter is used, the manufacturer's instructions are followed exactly.
- \_\_\_ 6. Standards are read in order from the lowest concentration to the highest concentration.
- \_\_\_ 7. 100 mL of sample or ammonia standard is measured into a 150 mL beaker.
- \_\_\_ 8. 1 mL of 10 N NaOH is added to all standards and samples to pH > 11 after electrode is immersed in the solution. (This prevents loss of ammonia gas).
- \_\_\_ 9. If more than 1 mL of 10 N NaOH is needed to raise the pH above 11, the volume is recorded and used in the calculations.
- \_\_\_ 10. Samples and standards are stirred slowly so that air bubbles are not sucked into solution.
- \_\_\_ 11. The electrode is allowed to stabilize before recording concentration or millivolt values.
- \_\_\_ 12. The electrode is rinsed between each standard and sample with ammonia-free water.
- \_\_\_ 13. The electrode is stored according to manufacturers guidelines.
- \_\_\_ 14. If distillation of samples is omitted, comparability data is on file for distilled vs. undistilled samples (The State Lab Study is acceptable). This does not apply to municipal effluent dischargers.

**TOTAL PHOSPHORUS, ASCORBIC ACID**  
**EPA METHOD 365.2 EVALUATION CHECKLIST**  
**(Methods 4500-P B(5) and 4500-P E, Standard Methods 18th ed.)**

Laboratory Name: \_\_\_\_\_ Lab Director: \_\_\_\_\_

Date: \_\_\_\_\_ Evaluator: \_\_\_\_\_

**I. EQUIPMENT**

- ☐ A. Spectrophotometer suitable for measurements at 880 or 650 nm, with a light path of at least 2.5 cm.
- ☐ B. Filter photometer equipped with a red color filter and a light path of 0.5 cm or longer.
- ☐ C. Hotplate or Autoclave for digesting samples.

**II. REAGENTS**

A. Digestion Reagents:

- ☐ 1. 11 N sulfuric acid,  $\text{H}_2\text{SO}_4$  for digesting samples made by diluting 300 ml of conc.  $\text{H}_2\text{SO}_4$  with 1 L of distilled water.
- ☐ 2. Ammonium persulfate,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , or potassium persulfate,  $\text{K}_2\text{S}_2\text{O}_8$ .
- ☐ 3. 1 N sodium hydroxide,  $\text{NaOH}$ .
- ☐ 4. Phenolphthalein indicator aqueous solution.

B. Method Reagents:

- ☐ 1. Potassium antimonyl tartrate solution:
  - ☐ Purchased commercially.
  - ☐ Made in laboratory by dissolving 1.3715 g  $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2} \text{H}_2\text{O}$  in 400 ml distilled water and diluting to 500 ml.
- ☐ 2. Ammonium molybdate solution:
  - ☐ Purchased commercially.
  - ☐ Made in laboratory by dissolving 20 g  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 1 \text{H}_2\text{O}$  in 500 ml distilled water.
- ☐ 3. Ascorbic acid, 0.1 M:
  - ☐ Purchased commercially.
  - ☐ Prepared weekly in lab by dissolving 1.76 g ascorbic acid in 100 ml distilled water.
  - ☐ Stored at 4° C.
- ☐ 4. Combined Reagent:
  - ☐ The above reagents are mixed in the following order and proportions for 100 ml of the combined reagent: 50 ml 5 N  $\text{H}_2\text{SO}_4$ , 5 ml of potassium antimonyl tartrate solution, 15 ml of ammonium molybdate solution, and 30 ml of ascorbic acid solution.
  - ☐ The reagents are at room temperature before they are mixed.
  - ☐ The solution is mixed after the addition of each reagent.
- ☐ 5. Stock phosphate solution (50 mg P/L, 1.0 ml=0.05 mg P)
  - ☐ Purchased commercially.
  - ☐ Prepared in lab by dissolving 219.5 mg anhydrous  $\text{KH}_2\text{PO}_4$  in dist. water and diluting to 1 L.
  - ☐ Stored at 4° C
  - ☐ Replaced as necessary
- ☐ 6. Standard phosphate solution (0.5 mg P/L, 1.0 ml=0.5 µg P)
  - ☐ Purchased commercially.
  - ☐ Prepared in lab by diluting 10.0 ml of stock phosphate solution to 1000 ml with dist. water.
  - ☐ Other concentration and/or preparation:(specify) \_\_\_\_\_
  - ☐ Stored at 4° C
  - ☐ Replaced as necessary
- ☐ 7. Working phosphate standards:

- \_\_\_ Prepared in laboratory by making serial dilutions of the standard phosphate solution.
- \_\_\_ Cover at least the concentration range of 0.01 to 0.50 mg P/L for low level samples and 0.1 to 1.0 mg P/L for wastewater samples.

### III. Procedure

#### A. Digestion

- \_\_\_ 1. 1 ml of 11 N H<sub>2</sub>SO<sub>4</sub> and 0.4 g ammonium or potassium persulfate are added to the appropriate volume of samples, standards, and blanks.
- \_\_\_ 2. Treated samples, standards, and blanks are boiled on a hot plate for 30-40 minutes or autoclaved for 30 minutes at 15-20 psi. Samples are not boiled dry.
- \_\_\_ 3. Samples, standards, and blanks are then cooled, diluted, adjusted to pH 7.0±0.2 and diluted to 100 ml.

#### B. Analysis

- \_\_\_ 1. 8.0 ml of combined reagent is mixed with 50.0 ml of samples, standards, and blanks.
- \_\_\_ 2. Color development is at least 10 min., but less than 30 min. before measuring absorbance or %T.
- \_\_\_ 3. Absorbance is measured at 880 or 650 nm.
- \_\_\_ 4. A reagent blank is used as the reference solution to set 0 Abs. or 100% T.
- \_\_\_ 5. A standard curve is prepared by plotting the absorbance values of at least 3 different standards and a reagent blank vs. concentration.
- \_\_\_ 6. The standard curve is linear (correlation coeff. >0.995), and passes through the origin.
- \_\_\_ 7. The standards bracket the concentration range of the samples.
- \_\_\_ 8. A blank and a standard are analyzed with each series of samples.
- \_\_\_ 9. Calculations are done correctly.
- \_\_\_ 10. Results are reported as mg P/L.

**TOTAL SUSPENDED SOLIDS, DRIED AT 103-105° C**  
**EPA METHOD 160.2 EVALUATION CHECKLIST**  
**(Method 2540 D, Standard Methods, 18th ed.)**

Laboratory Name: \_\_\_\_\_

Lab Director: \_\_\_\_\_

Date: \_\_\_\_\_

Evaluator: \_\_\_\_\_

**I. EQUIPMENT**

- ☐ A. Drying Oven, for operation at 103 to 105° C or,
- ☐ B. Analytical Balance, capable of weighing to 0.0001 gram
- ☐ C. Desiccator
  - ☐ active colored desiccant indicates that the drying capacity is not exceeded.
  - ☐ desiccator cover is sealed tightly.
- ☐ D. Glass-fiber filters used:
  - ☐ Whatman 934AH
  - ☐ Whatman 984H
  - ☐ Gelman A/E
  - ☐ Millipore AP40
- ☐ E. Filter Holder, Gooch crucible adapter or membrane filter funnel.
- ☐ F. Gooch crucible, 25 to 40 ml capacity, suitable for filter size selected.
- ☐ G. Suction flask, of sufficient capacity for sample size used.
- ☐ H. Weighing dishes, if membrane filter funnel is used.
- ☐ I. Tongs or forceps
- ☐ J. Vacuum source

**II. Procedure**

**A. Preparation of Glass Fiber Filters**

- ☐ 1. Filters are seated with wrinkled side up.
- ☐ 2. Filters are washed with 3 successive 20 ml portions of distilled water under vacuum.
- ☐ 3. Filters are dried in oven at 103 to 105° C for at least 1 hour.
- ☐ 4. After drying, filters (and gooches or weighing dishes) are stored in desiccator until cool.
- ☐ 5. Balance is zeroed before weighing filters.
- ☐ 6. Filters (and gooches or weighing dishes) are weighed before use.

**B. Sample Treatment**

- ☐ 1. Samples are well mixed and unrepresentative particles avoided when measuring volumes.
- ☐ 2. Samples are filtered under vacuum.
- ☐ 3. Sample volumes used yield no more than 200 mg total suspended solids.
- ☐ 4. The solids capture is 1 mg, if 4.7 cm filters are used.
- ☐ 5. After samples are filtered, filters are washed with 3 successive 10 ml portions of distilled water, dried (see #8 below), and cooled before weighing.
- ☐ 6. Balance is zeroed before weighing residue.
- ☐ 7. Gooches or weighing dishes are handled with tongs.
- ☐ 8. Samples are filtered so that plugging of filter is prevented.
- ☐ 9. Samples are dried overnight, or drying cycle is repeated:
  - ☐ Until constant weight is attained, or
  - ☐ Until weight loss is less than 4% of previous or 0.5 mg, whichever is less, or
  - ☐ On occasion to check drying efficiency. Frequency: \_\_\_\_\_
- ☐ 10. Drying time is determined after the oven is up to temperature.

NOTE: DAILY BLANKS ARE NOT REQUIRED IF SAMPLES ARE DRIED OVERNIGHT

## QUALITY CONTROL CHECKLIST: TEST CATEGORIES 1-4:

Laboratory Name: \_\_\_\_\_ Laboratory Director: \_\_\_\_\_

Date: \_\_\_\_\_ Evaluator: \_\_\_\_\_

Category/Analyte:	BOD 5	TSS	Ammonia Nitrogen	Phos-phorus	Nitrate Nitrogen	TKN Nitrogen	Total Solids	TDS	Volatile Solids	CBOD 5
Are reagent blanks analyzed each analysis day?		NA					NA	NA	NA	
Are duplicate samples run after the analyses of 10 samples?										
Are spiked samples analyzed after the analyses of 20 samples?	NA	NA					NA	NA	NA	NA
Is the method of standard addition used instead of spiking samples?	NA	NA					NA	NA	NA	NA
If more than 20 samples are analyzed in 1 day, is a known standard analyzed after every 20 samples?	NA	NA					NA	NA	NA	NA
Is the instrument response for the known standard after 20 samples within $\pm 10\%$ of the calibrated value?	NA	NA					NA	NA	NA	NA
Is a known standard analyzed once per week or after the analysis of 20 samples?		NA	NA	NA	NA	NA	NA	NA	NA	
If the result of a known standard exceeds QC limits, does the lab take corrective actions?		NA					NA	NA	NA	
Is one blind standard analyzed every 3-5 months if the analyte was analyzed during the previous 3-5 months?										
Are QC limits established for duplicate samples?										
Is the method for calculating precision control limits the same as the method states?										

NA= Not Applicable

Note: There are special requirements for labs analyzing more than 20 samples in a batch. See the DNR QA plan for additional requirements.

## QUALITY CONTROL CHECKLIST: TEST CATEGORIES 1-4:

Category/Analyte:	BOD 5	TSS	Ammonia Nitrogen	Phosphorus	Nitrite Nitrogen	TKN Nitrogen	Total Solids	TDS	Volatile Solids	CBOD 5
Does the laboratory use its own duplicate data to calculate its own control limits? If not, explain.										
Are calculations for control limits correct?										
Does the lab take corrective action when a duplicate sample exceeds QC limits?										
If a duplicate QC limit is exceeded and a discrepancy has affected past sample results, are samples reanalyzed or the results qualified back to the last acceptable QC check?										
If a known standard QC limit is exceeded and a discrepancy has affected past sample results, are samples reanalyzed or the results qualified back to the last acceptable QC check?										
Are QC limits established for spiked samples?	NA	NA					NA	NA	NA	NA
Does the laboratory use its own spike data to calculate its control limits? If not, explain.	NA	NA					NA	NA	NA	NA
If a spiked sample QC limit is exceeded and a discrepancy has affected past sample results, are samples reanalyzed or the results qualified back to the last acceptable QC check?	NA	NA					NA	NA	NA	NA
Is documentation available for corrective actions taken to bring QC results back within limits?										
Does the laboratory have a QA plan? Do they follow it?										
Is a copy of the methodology available to the analysts?										

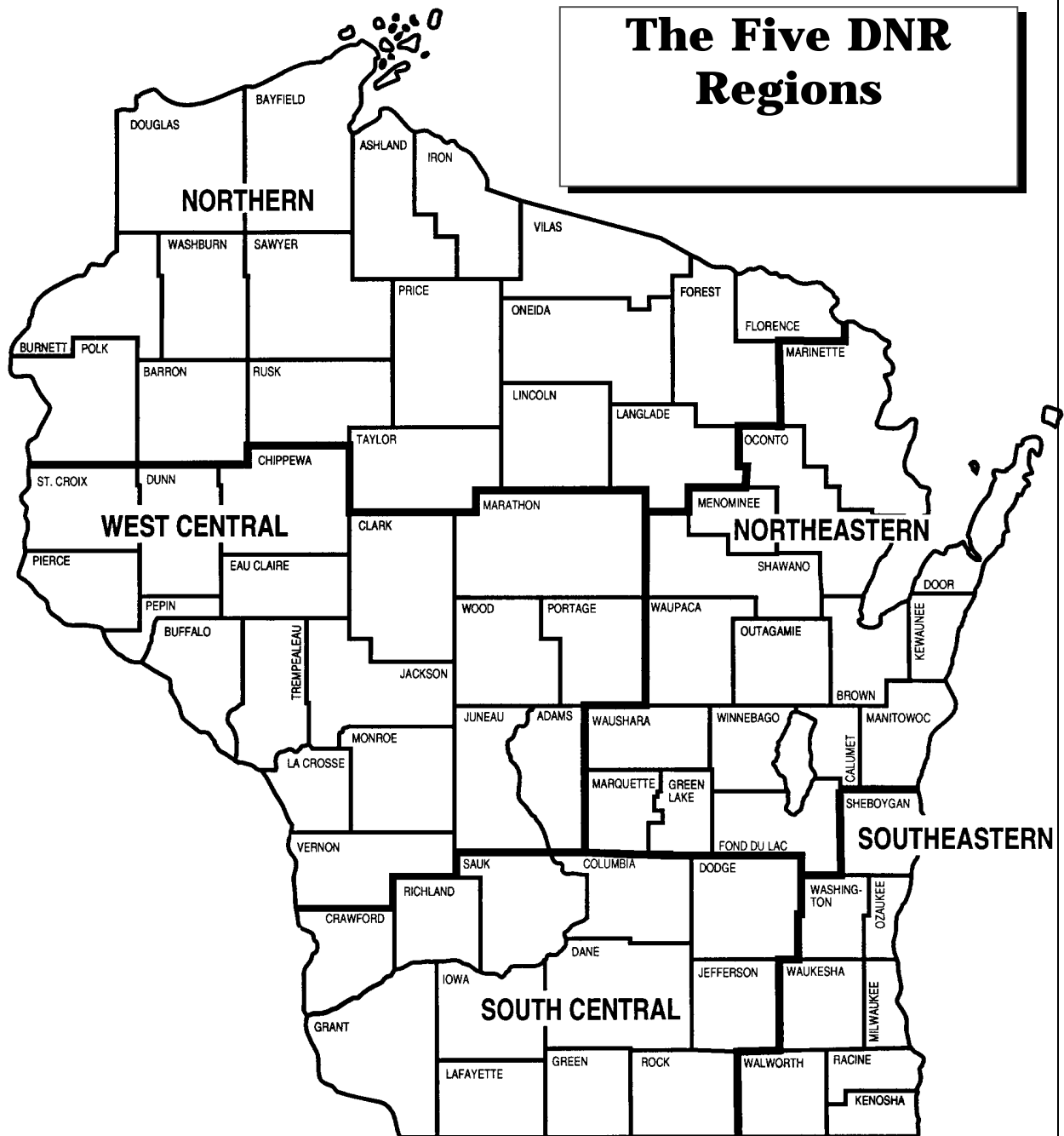
NA= Not Applicable

Note: There are special requirements for labs analyzing more than 20 samples in a batch. See the DNR QA plan for additional requirements.

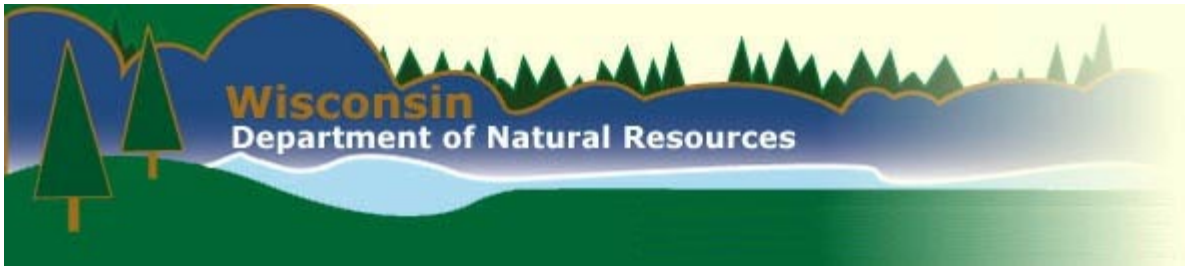
### QUALITY CONTROL CHECKLIST: TEST CATEGORIES 1-4:

Category/Analyte		BOD	TSS	Ammonia Nitrogen	Phosphorus	Nitrate Nitrogen	TKN Nitrogen	Total Solids	TDS	Volatile Solids	CBOD
Can a record of samples processed be traced back to the:	Analyst										
	Date										
	Method Used										
Is information on the maintenance of lab instruments retained? List equipment and explain if necessary.											
Has the DNR requested that these records be kept longer than three years? Are they?											
If an alternate methodology or variation of a method is used, is this documented and approved?											

## The Five DNR Regions







# DNR Mission Statement

To protect and enhance our Natural Resources -  
our air, land and water;  
our wildlife, fish and forests.

To provide a clean environment  
and a full range of outdoor opportunities.

To insure the right of all Wisconsin citizens  
to use and enjoy these resources in  
their work and leisure.

And in cooperation with all our citizens  
to consider the future  
and those who will follow us.